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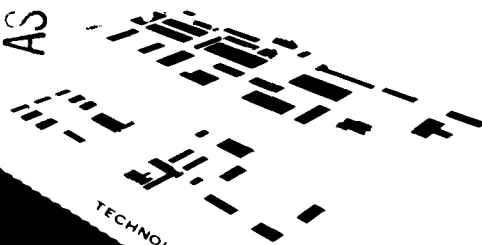
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Third Quarterly Report  
April 1963

GLASS FIBER STRENGTH ENHANCEMENT  
THROUGH BUNDLE DRAWING OPERATIONS

for

Bureau of Naval Weapons  
Navy Department  
Washington, D. C.

Prepared under

Contract No. N600(19)58450

**ARMOUR RESEARCH FOUNDATION**  
of  
**ILLINOIS INSTITUTE OF TECHNOLOGY**  
Technology Center  
Chicago 16, Illinois

**GLASS FIBER STRENGTH ENHANCEMENT**  
**THROUGH BUNDLE DRAWING OPERATIONS**

**Third Quarterly Progress Report**

Compiled by R. H. Cornish  
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for  
**Bureau of Naval Weapons**  
**Navy Department**  
**Washington, D. C.**

**Prepared under**  
**Contract No. N600(19)58450**

## FOREWORD

This is the third quarterly progress report on Armour Research Foundation (ARF) Project K263 (ARF Report No. 8263-3) covering the period of work on Contract No. N600(19)58450 from December 7, 1962 to March 7, 1963. This contract is under the direct supervision of the Non-Metals Branch, Materials Division, Bureau of Naval Weapons with Mr. M. Stander RRMA-32 acting as BuWeaps technical monitor.

ARF staff members who have contributed to the research discussed in this report include Richard M. Chaney, Kenneth Coleman, Dr. Rodney H. Cornish, Joseph S. Islinger, Erich Koeller, Dr. David L. Levinson, H. Robert Nelson, James T. Staulcup and Dr. Nicholas A. Weil.

Experimental data on this project are recorded in ARF Logbooks No. C12168 2nd, C12177.

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION OF  
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## GLASS FIBER STRENGTH ENHANCEMENT THROUGH BUNDLE DRAWING OPERATIONS

### 1. INTRODUCTION

This program is a continuation of a previous Armour Research Foundation program completed under Contract No. NOW61-0259. The objective of the previous program, which ran from January 1961 to April 1962, was the investigation of methods capable of producing glass fibers with enhanced tensile strengths. Most of the work was based on the "bundle drawing" concept which was expected to yield flaw-free submicron diameter fibers. Results of the previous work were summarized in the final report on ARF Project K213 issued in May 1962.

Included in the final report was an analytical derivation which predicted that no fibers below a certain diameter could be drawn in a monofilament operation. In the various phases of the program, work included the drawing of several glasses from rod form into fibers, the subsequent protective coating of such fibers with metals and the bundle drawing of these fibers to establish feasibility. A technique was developed for applying metallic coatings to "as-drawn" virgin fibers. Of the alloys explored in the original program 80 Pb-20 In and 95 Al-5 Si appeared to show the most promise from the standpoint of wetting the glass. Unfortunately those alloys which coated readily at temperatures well below the softening point of the glass dewetted readily at the glass softening temperature. This dewetting allowed the individual fibers to fuse during bundle drawing. As a result redrawing of bundled metal coated fibers was not achieved. However, the feasibility of bundled redrawing as a means for producing fine filaments was demonstrated using a glassy matrix.

As a result of the promising results obtained with a glassy matrix to separate the fibers, a continuation phase of this research program was authorized with the following major objectives:

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- (1) Studies of the dependence of the strength of glass fibers upon their dimensional characteristics principally as represented by the fiber diameter.
- (2) A comprehensive investigation of the wettability and soundness (adherence and continuity) as well as the dewetting characteristics of metallic coatings applicable to glass.
- (3) Studies in the metallizing of fibers, including a study of parametric effects (speed of drawing, fiber size, metallic system) upon the resulting thickness, uniformity, and continuity of coating.
- (4) Establishment of the feasibility of bundle drawing operations and exploration of the parameters conducive to an optimization of such a process.

Progress toward these four goals as reported in the First and Second Quarterly Reports on ARF Project K263 was:

- (1) Development of handling and mounting techniques for virgin fibers coupled with establishment of the basic requirements for a fiber tester plus the design and construction of such a device.
- (2) Study of wetting - dewetting characteristics of a new set of alloys doped with Ti and/or Zr. This set of alloys was found to resist dewetting at temperatures well above their wetting (minimum coating) temperatures and into the glass softening range.
- (3) Metallizing techniques extended to drawing of premetallized glass rods both with and without outer glass sheaths to produce premetallized glass fibers as well as glass - metal - glass composite fibers. Studies of the fiber coating immediately after drawing were continued and expanded to include the new alloy systems. The metal -

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lizing apparatus developed for use in the prior program was refined to facilitate the production of symmetric and metallurgically sound coatings.

- (4) Investigation of the fundamental parameters for successful bundle drawing included several combinations of glass rods in metal matrices enclosed within a suitable sheath. Conditions for the drawing of metal coated glass fibers in simple geometries were established.

This report discusses progress during the third quarterly reporting period. Progress in the individual areas during this period may be summarized as follows:

- (1) The glass fiber tester and its accessory enclosure were completed to facilitate drawing, capturing, and testing of virgin fibers in a controlled environment. The test schedule for the strength studies to be carried out during the next period was set up.
- (2) Wetting - dewetting studies on the candidate alloy coating materials were temporarily slowed to await feedback of information from the metallizing and bundle drawing studies.
- (3) Investigation of metallizing techniques utilizing both pre-coating and post - draw - coating were continued, using the previously reported Titanium and Zirconium doped alloys, deposited coating of the noble metals, and some work with drawing of glass - Kovar - glass and glass - aluminium glass systems.
- (4) Bundle drawing investigations were extended to include sheathed and sheathless bundles of glass - metal - glass composite fibers and soft glass coated glass fibers in addition to continued work with the metallic matrices. Bundles of round and semi-elliptical submicron fibers were successfully drawn using both the glass - glass and glass - metal glass combinations in sheathed and sheathless configurations. Dewetting continues to plague efforts to redraw in a metallic matrix. Initial conditions for the systematic drawing of bundles of submicron fibers have been established.

(5) As an addendum to the bundle drawing investigations research has begun into methods for removing glassy matrix material from the redrawn bundle. This step is necessary for the glassy matrices in order to assess the strength enhancement realizable in the bundle drawing operation.

Progress in these areas is delineated in more detail in Section II of this report.

## II. DISCUSSION

### 1. Strength Studies

During this research period the modifications to the fiber drawing apparatus to permit fiber drawing and testing in an inert atmosphere were completed. The fiber tester installed in the inert gas drawing and testing enclosure is shown in Figure 1.

The gas-tight enclosure facilitates evacuation and/or purging of the atmosphere surrounding the working parts of the fiber drawing apparatus and fiber tensile tester. These provisions have been made so that the effects of humidity and other deleterious agents inherent in the atmosphere can be minimized for both the drawing and subsequent strength experiments.

A series of investigations of the effect of fiber diameter upon strength using these closely controlled fiber drawing and testing conditions is planned for the next research period. E glass fibers in several diameters will be drawn from rods under as identical conditions as practical in an inert (nitrogen gas) atmosphere. In order to produce fibers of several diameters the factor to be varied will be the drawing speed. Some fibers of each diameter will be collected by winding upon the drum while others will be captured as virgin fibers. The virgin fibers will be tested for tensile strength in the inert atmosphere immediately after drawing. The drum wound fibers will then be divided into two groups. Some will be tested in the inert atmosphere while the others will be stored under normal laboratory conditions and, should time permit, will be tested under similar conditions.

It is expected that a maximum of three virgin fibers will be available for testing from any one draw, for as soon as the virgin fibers are captured a new draw must be started and stable drawing conditions re-established. Nevertheless every effort will be made to test an adequate number of such virgin fibers to permit a statistical analysis of the tensile strength data.

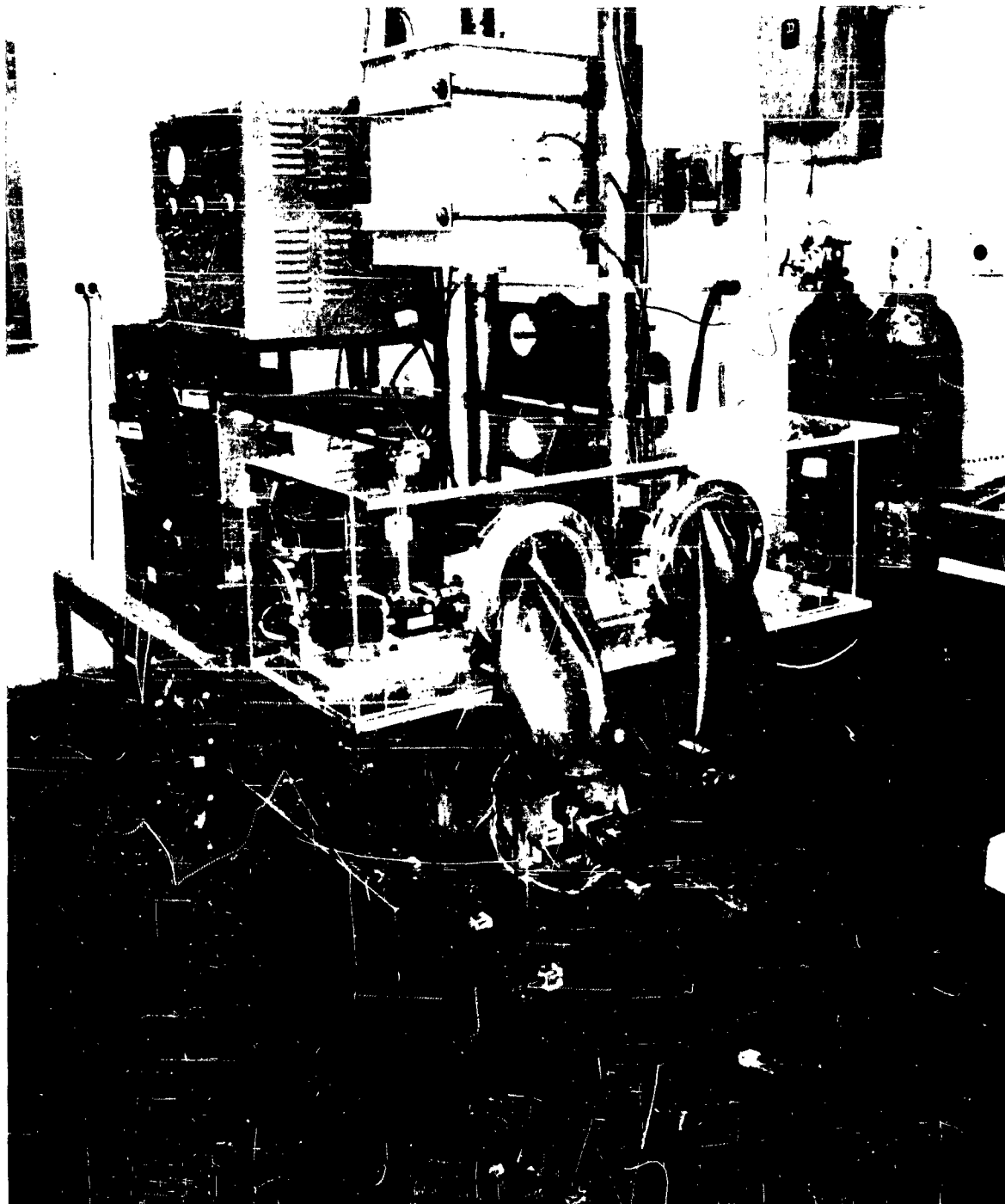


Figure 1

Controlled Atmosphere Drawing And Testing Enclosure

## 2. Wetting - Dewetting Studies

Research investigations of the last quarter have been confined to variation of the Titanium and Zirconium contents used to formulate alloys for use in the metallizing studies. In the last quarter of this program compilation of wetting - dewetting data established during the program will be completed. Existing data will be augmented if feedback of information from the metallizing and bundle drawing studies indicate a need for additional alloys.

## 3. Metallizing Studies

Metallizing investigations during the last quarter as during previous quarters have been directed toward the development of a technique or techniques for applying continuous, uniform, well bonded and metallurgically sound metallic coatings to glass fibers. The ultimate purpose remains the establishment of parameters for metallizing fibers preparatory to bundling and redrawing. As reported in the last quarterly report, the magnitude of the problem associated with applying coatings which wet readily and at the same time resist dewetting during redrawing has resulted in the evolution of several parallel approaches to the problem.

The first approach involves refinement of the post draw metallizing procedures used in the previous program. This procedure consists of pulling a fiber (in this case, directly out of the drawing furnace) through a suitably supported melt of the coating material. The second technique is based on drawing of a precoated rod into fiber form to produce a uniformly metallized fiber. This technique is also applicable to the production of fibers coated with both metal and soft glass and of fibers coated with glass alone. In addition to the previous approaches post-draw coating with resinate solutions of noble metals followed by sintering of the coating has been explored. These three approaches are discussed separately in the following paragraphs.

Coating during drawing is very attractive from the standpoint of basic simplicity and the fact that a protective coating can be applied to the fiber immediately after its emergence from the drawing furnace. During the second quarter a refined metallizing apparatus was developed. In this device, a melt of the coating material is contained in a resistance

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heated crucible and feeds through an orifice in the crucible wall to form a meniscus of molten metal. Metallizing is effected by drawing the fiber through this meniscus. To mechanically stabilize the meniscus and to reduce the possibility of abrading the virgin fiber against the crucible face, a moveable heated plate has been added in front of the crucible orifice. By proper positioning and temperature control of this plate, it has been possible to establish a relatively thick and stable meniscus. During the third quarter, which is reported here, this modified unit was used principally to coat virgin fibers with Ti and Zr doped 80 Pb - 20 In alloy. Fibers were not successfully coated using the Easyflo alloy by this technique. It became apparent that the existing heater would not heat the alloy enough above its melting point ( $1125^{\circ}\text{F}$ ) to permit sufficient flow through the crucible orifice. A new heater has been fabricated and will be used in subsequent efforts to coat with the doped Easyflo system.

The results of fiber coating investigations using the doped 80 Pb - 20 In alloy have been very promising from the standpoint of applying sound symmetric and uniform coatings. Not enough tests have been completed within the reported period to completely establish the parametric condition necessary to obtain a preselected coating thickness and quality for a given fiber diameter and drawing speed. It has, however, been clearly established that a broad range of coating thicknesses can be attained. Examples of the cross sections of metallized E glass fibers are shown in Figs. 2, 3 and 4 to illustrate the attainable thickness control, symmetry, and soundness of fiber coatings.

Evaluation of the quality of the metallized 80 Pb - 20 In coatings has proceeded according to the three tests proposed in the last report. These tests included, microscopic examination, evaluation of the resistance to chemical attack and effectiveness as a matrix agent in re-drawing operations. Microscopic inspection of the lateral surface under a low power eyepiece showed longitudinal continuity which was further verified by checks of electrical resistances along the coated fiber. Boiling in aqueous NaOH indicated that the coated fibers would remain intact at least 10 times as long as uncoated fibers. Redrawing of the coated fibers as reported in

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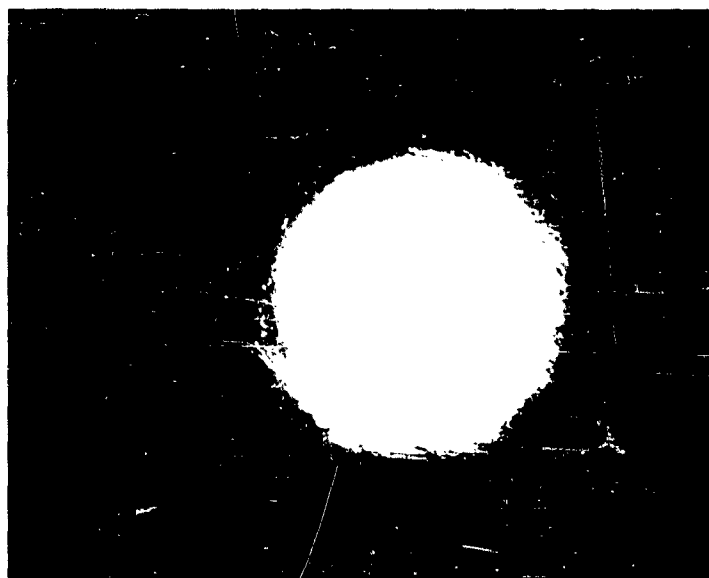


Etchant: 5% HF

X600

Figure 2

"E" Glass Fiber Coated with Ti/Zr Doped  
80 Pb - 20 In Alloy. Fiber Diameter 45  $\mu$   
Coating Thickness 5  $\mu$

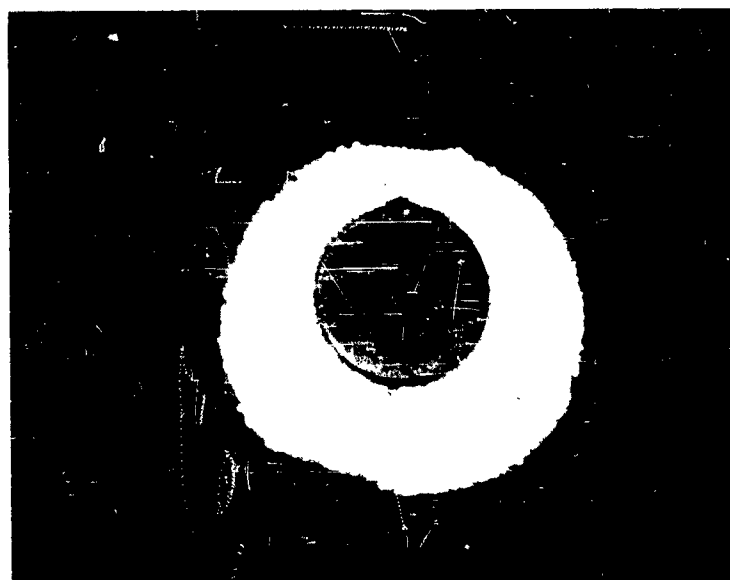


Etchant: 5% HF

X600

Figure 3

"E" Glass Fiber Coated With Ti/Zr Doped  
80Pb - 20 In Alloy. Fiber Diameter 45  $\mu$   
Coating Thickness 11  $\mu$



Etchant 5% HF

X600

Figure 4

"E" Glass Fiber Coated With Ti/Zr Doped  
80Pb - 20 In Alloy. Fiber Diameter 45  $\mu$   
Coating 25  $\mu$

the next subsections have not as yet shown this coating to be satisfactory from the standpoint of providing a satisfactory redraw matrix.

The precoating approach to fiber metallizing has been continued along three broad lines. The first of these is based on the gas protected immersion technique described in the last progress report. In this approach, the metallizing alloy is placed in the bottom of a closed sheath which has a tee-type connection at the top. The sheath is evacuated; the alloy melted by external heating of the sheath; the system flooded with argon gas and the degreased rod to be coated, preheated and inserted into the melt. The alloy is forced up around the tube coating the fiber. The resulting composite can be slowly cooled to produce a glass - metal - sheath system or the coated rod can be removed from the sheath before freezing to produce a simply metallized rod. This technique is applicable to thicknesses of from 0.002 in. to 0.050 in. consisting of either the doped Easyflo or doped 80 Pb - 20 In alloys.

Efforts to extend this technique to the precoating of bundles of rods have met with mixed success. The use of 80Pb - 20 In in this process was quite straightforward. The substantially higher melting point of the Easyflo system coupled with the lower ratio of cross-sectional to surface area for the bundles of rods results in excessive twisting of the rods during insertion into the melt which makes it virtually impossible to produce composites useable for drawing.

In addition to work with the doped Easyflo and 80Pb - 20 In alloys modifications of this system has been evolved to use deposited and fired on coatings as precoating agents as well as thin sheets of aluminum and Kovar. Application of the organically deposited and sintered coatings is quite straightforward. The E glass rods to be coated are degassed, and if necessary polished with agitated 5% HF to remove surface contamination. The suspended coating is then brushed on, the carrier boiled off and the metal fired onto the glass using manufacturer recommended temperature cycles. The coated rods can then be drawn freely or within a glass sheath. A summary of the experiments which have been conducted using this coating procedure is given in Table 1. Both aluminum and Kovar have been applied by wrapping foil around an E glass rod and enclosing this in a soft glass

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TABLE 1  
SUMMARY OF PRECOATING EXPERIMENTS ON "E" GLASS RODS

Coating Material	Method of Application	Number of Experiments	Thickness	Coatability	Drawability
Platinum	Resinate Deposited	6	<10 $\mu$	Excellent	Interrupted
Gold	Resinate Deposited	15	<10 $\mu$	Excellent	Good
Gold	Resinate Deposited	4	>10 $\mu$	Excellent	Good (Deteriorates Fibers)
Silver	Resinate Deposited	4	35 $\mu$	Excellent	Good (Deteriorates Fibers)
Aluminum	Wrapped Foil	2	.003 in.	Poor	Interrupted
Aluminum	Tube	1	.030 in.	Poor	---
Kovar	Wrapped Sheet	2	.005 in.	Poor	---

sheath. The sheath was then evacuated and heated in a muffle furnace until the sheath collapses down onto the wrapped foil. The results of this approach are also summarized in Table 1.

As noted, the organically deposited layers of platinum and gold have demonstrated the best soundness, symmetry and structural adherence. Efforts to redraw the platinum coated E glass rods both with and without external sheathing have not been successful. Even though the platinum in both cases, remained intimately bonded to the glass, it did not have sufficient ductility at glass drawing temperatures to follow the glass deformations during drawing. The resulting fibers rather than being covered with a continuous layer showed small platelets of relatively thick platinum adhered over the entire surface. This effect is present regardless of coating thickness over the entire range of thicknesses applicable to the rod in the precoating operation. Admittedly smaller thickness might reduce in a uniform manner. Unfortunately, the thickness of coating needed on the ultimately redrawn fiber will not permit a precoating thickness less than 0.002 in. due to the draw and redraw processes which reduce the initial rod diameter by a factor of 2000 to 5000.

Gold applied by a similar process has been drawn in a sheathed configuration to produce an E glass fiber - gold - glass sheath composite with a uniform appearance. Composite fibers produced in this manner have been used extensively in the redrawing operations reported in the next subsection. Efforts to draw gold coated E glass rods into metal coated rods without the use of a sheath have not been successful as stable drawing conditions could not be established. The gold when drawn without a sheath cracks slightly in the area immediately preceding the drawing zone. In the case of drawing in a glass sheath microcracks did not appear, probably as a result of some lateral support from the sheath. At any rate, whenever these cracks appeared, the gold covered areas appeared to be shielded from radiant or convective heating with the result that the smooth reduction from rod to fiber was replaced by a corrugated section. The resulting fibers were quite lumpy and unapplicable to further drawing operations. Silver deposited by this method partially dewetted during attempted

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drawing from the coated rod whether or not a sheath is used. In addition to this partial dewetting the remaining silver reacted with the glass surface producing a weak fiber.

Both aluminum and Kovar vacuum sandwiched between an E glass rod and sheath could not be drawn successfully. The aluminum melted and dewetted during drawing and eventually accumulated in quantities sufficient to break the glass sheath. At the drawing temperatures Kovar would not follow the glass's reduction of area and caused fracture of the composite at approximately 20% reduction of area.

Since the resinate deposited and fired coatings in general bonded quite well to E glass rods preparatory to drawing, it was decided that experimentation with coating of the as-drawn fiber might be worth-while. On the basis of the work with precoated rods, this investigation was confined to gold. A virgin fiber from the drawing furnace was pulled through a simple bushing containing the coating liquid. Since the fiber was still warm when it reached the bushing, a large portion of the carrier in the resinate solution was boiled off as the fiber passed through the bushing. The emerging coated fiber was then heated with a pair of radiant heat lamps to remove the remaining carrier. These fibers were then either fired on an individual basis or bound together and fired for subsequent bundle drawing experiments.

This technique produces a uniformly coated fiber. Unfortunately, the surfaces of the individual fibers were degraded in this process.

Both the drawing of precoated rods and the metallizing of as-drawn fibers will be continued in support of the bundle drawing operations. The controlled atmosphere capability will be used as much as possible to improve both coating quality and the spectrum of coatings applicable to the precoating process.

#### 4. Bundle Drawing Studies

Bundle drawing investigations in the third quarter have been devoted to the redrawing of E glass fibers coated with, metals alone, metal plus a glass and glass alone. Efforts to draw bundled metal coated fibers in suitable sheaths has been continued using modified alloy systems as these become available from the wetting - dewetting and metallizing phases of the program. A significant amount of effort has been devoted to establishing the drawing parameters for producing sound low micron and submicron fibers from bundled glass - metal - glass and glass - glass composite fibers. The drawing experiments carried out during the third quarterly period are summarized in Table 2.

The redrawing of bundles of metallized E glass fibers has still not been achieved. Even though both metallic coatings employed in the reported research, wet very well and resist dewetting relatively well for short periods of time, the time at temperature in redrawing and the lateral forces tending to force the fibers together have to date resulted in fused fibers with segregated areas of metal as shown in Fig. 10 and 11. This is true not only for the sheathed redraws of Ti and/or Zr doped 80Pb - 20 In coated fibers but also for the E glass fibers coated with resinate deposited gold. The efforts to redraw lead-indium coated fibers have all used bundles of a few hundred metallized fibers placed into a sheath and fused to the sheath at one end for handling. Both relatively hard pyrex, and soft 1720, sheaths have been used without success. Dewetting takes place prior to final reduction as evidenced by balling up of the coatings on individual fibers. It has not yet been clearly established whether this is due to Ti and Zr forming their respective oxides from atmospheric oxygen or from available oxygen in the glass at drawing temperatures. Studies run with glass sheaths and bundled metallized fibers evacuated prior to drawings have shown roughly similar results. The planned drawing experiments contained wholly within an inert atmosphere should resolve this question.

As indicated by Table 2 most of the effort in this phase of the program during the last quarterly period has been devoted to sheathed and sheathless redrawing of bundled glass - metal - glass and glass - glass composite fibers. Drawing of glass - on glass fibers was reported in the final

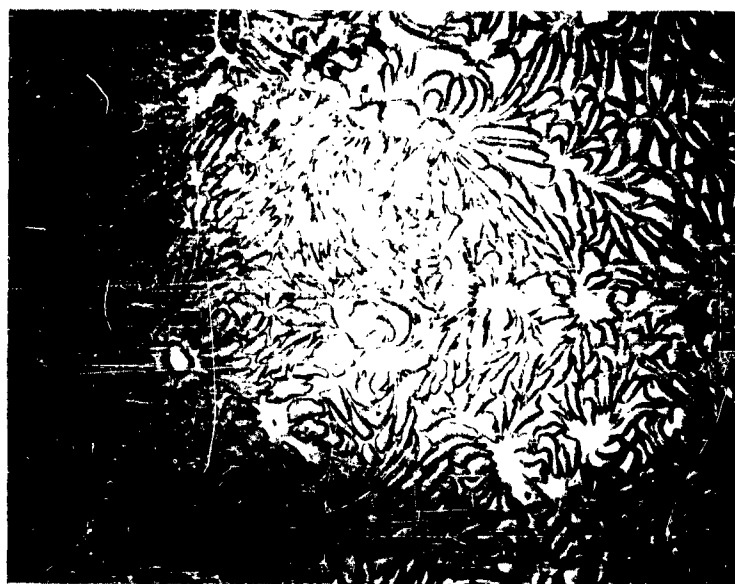
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TABLE 2

SUMMARY OF BUNDLE DRAWN "E" GLASS FIBERS

Matrix Material	Metal Interface Material	Proportion of Glass Coating Cross-Sectional Area to "E" Glass Cross Sectional Area	Sheathed*or Unsheathed Bundle	Reduction of "E" Glass	Mean Furnace Temp.	Shape of "E" Glass Fiber	Figure No.
Pyrex	Gold	1:1.9	Sheathed	7:1	2100°F	Flat	7
Pyrex	Gold	1:1.9	Unsheathed	6:1	2100°F	Flat	6
7052	None	1:2.3	Unsheathed	50:1	1900°F	Round	7
7052	None	1:2.3	Sheathed	52:1	2000°F	Round	-
7052	Gold	1:2.1	Unsheathed	48:1	2000°F	Round	-
7050	Gold	1:2.1	Sheathed	47:1	1950°F	Round	8
1720	None	1:1.8	Sheathed	Fused	2150°F	Flat	-
1720	Gold	1:1.8	Unsheathed	Fused	2050°F	Flat	11

\*All sheath materials are 1720 glass.



Etchant: 5% HF

600X

Figure 5

Bundle Drawn "E" Glass Fiber In Pyrex Matrix  
With Gold Interface and 1720 External Sheath



Etchant: 5% HF

600X

Figure 6

Bundle Drawn "E" Glass Fiber In Pyrex Matrix  
With Gold Interface And No External Sheath

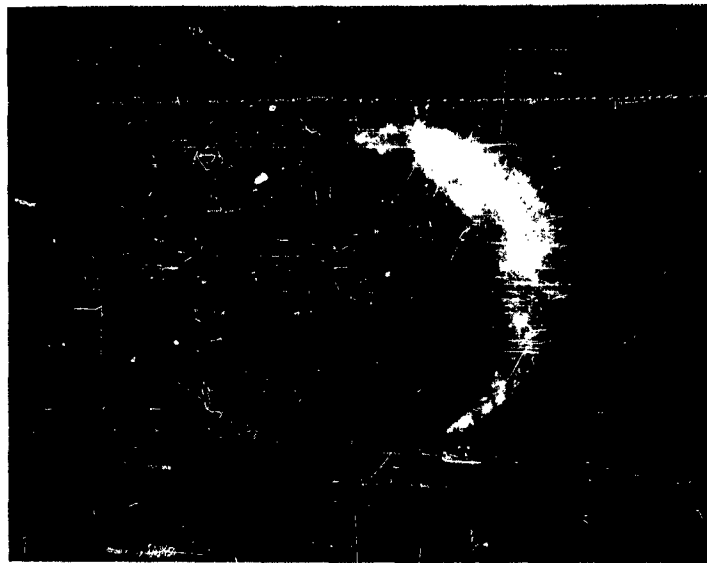


Etchant: 5% HF

600X

Figure 7

Bundle Drawn "E" Glass Fiber In 7052 Matrix  
With No Metal Interface and No External Sheath



Etchant: 5% HF

600X

Figure 8

Bundle Drawn "E" Glass Fiber in 7050 Matrix  
With Gold Interface and 1720 External Sheath

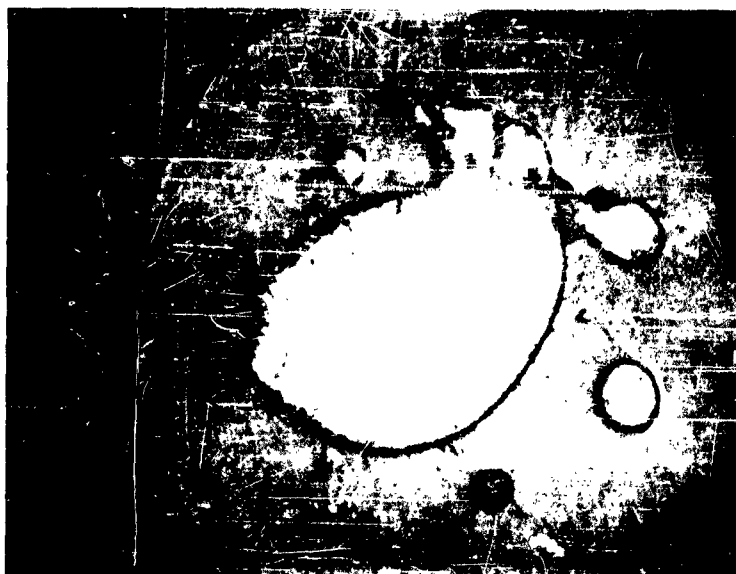


Etchant: 5% HF

600X

Figure 9

Bundle Drawn "E" Glass Fiber in 1720 Matrix  
With Gold Interface and 1720 External Sheath

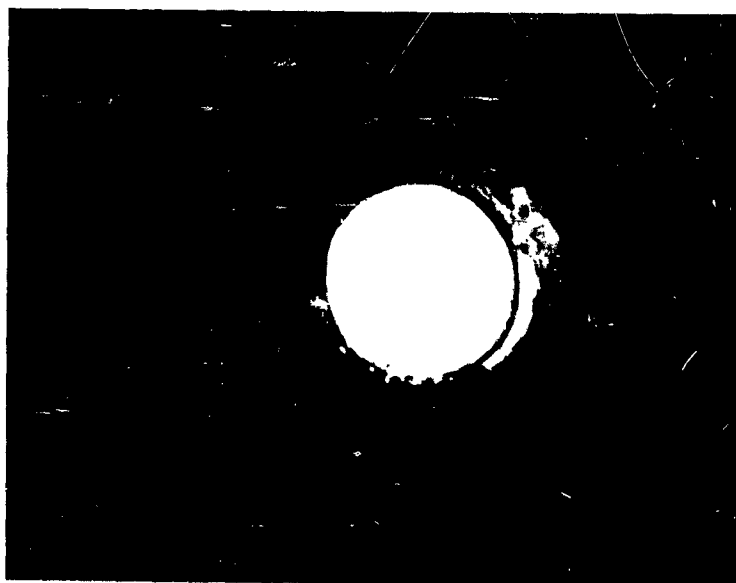


Etchant: 5% HF

600X

Figure 10

Redrawn Bundle of Ti/Zr Doped 80Pb-20 In Coated  
"E" Glass Fibers. Metal Dewetted and Fibers Fused



Etchant: 5% HF

600X

Figure 11

Redrawn Bundle of Resinate Deposited Gold Coated  
"E" Glass Fibers. Metal Dewetted and Fibers Fused.

report of a prior program. The success attained with glassy matrices has led to the investigation of glass fibers both with and without a metallic coating drawn within a glassy matrix. In this effort bundles consisting of several hundred glass coated fibers or glass - metal - glass composite fibers were redrawn to form bundles of micron and submicron fibers encased in a glassy matrix. Two broad techniques for redrawing were employed. In the first approach the fibers were packed into a suitable sheath as in the case of prior work with metallized fibers. In the second approach the fibers were drawn without a sheath using only fusing at the end to join the fibers.

During the draw process the outer glass coatings fuse to form a continuous matrix for the redraw. Initial efforts at redrawing used a pyrex sheath. The high temperature required for drawing of the pyrex fiber coating and/or sheath was sufficient to cause the E glass to deform into hexagonal cross sections to fill the voids inherent in packing of the bundle. This effect is shown in Figure 5, and is apparent for bundled fibers drawn with as well as without external sheathing. On the basis of this evidence and prior predictions it was decided to look into softer glasses for matrix materials. As was expected these matrices can be used to produce bundles of round submicron fibers as shown in Figures 7 and 8 for glass - glass and glass - metal - glass composite fibers respectively. Redrawing in a glassy matrix does produce some anomalies depending on drawing temperatures, initial bundling, packing and material combinations. First of all, if significant voids exist in the bundle prior to redraw, the matrix plus fibers will not pack uniformly and the redrawn fibers plus matrix will tend to flow into the void producing elliptical fibers in the region surrounding the void. Careful packing, softer sheaths, and higher drawing temperatures reduce this tendency. A second effect that has been noted primarily in the redrawn bundled glass - metal - glass composites consists of the fibers and matrix fusing into a round bundle and migrating to one side of the sheath, as evidenced in Figure 8. This effect is not as prevalent in glass on glass giving some credence to a differential heating effect between the sheath and bundle. The second effect just as the first can be eliminated by using a soft external sheath and relatively high drawing temperatures.

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Bundles of submicron and micron fibers produced from both glass - on glass and glass - metal - glass composites have been successfully produced. Figures 7 and 8 show the best examples, to date, of fibers drawn by this technique.

It is convenient that the soft glass matrix and sheath materials produce the most uniform fibers as certain glasses in this class can be etched at very high rates compared to E glass and thus lend themselves to bundle separation studies planned for the next quarter.

### III PLANS FOR FUTURE WORK

Future work on this program will be aimed toward developing a technique for realistically demonstrating the enhanced strength potential of bundled submicron fibers. The basic breadth of the program will be preserved in order to support further research efforts and correlate the results of strength tests on submicron size fibers with micron and commercial size fibers. Work in the specific problem areas will proceed as follows:

#### 1. Strength Studies

The completed fiber tester and inert gas enclosure will be used to evaluate virgin and drum wound fibers without exposing them to atmospheric degradation. In addition drum wound fibers will be stored under normal laboratory conditions and if time allows tested after predetermined exposure times.

#### 2. Wetting - Dewetting Studies

Studies in this area will be primarily confined to further compilation of data on the wetting and the kinetics of dewetting for the candidate alloy systems doped with Ti and/or Zr. If time permits further work on the dewetting characteristics of deposited and fired noble metal coatings will be carried out.

#### 3. Metallizing Studies

Work will continue on metallizing during drawing to establish critical operating parameters for the modified metallizing unit described in the second quarterly report. In addition to this, efforts to continuously deposit and sinter noble metal coatings will continue. If time permits, the inert atmosphere drawing capability will be used to further extend the spectrum of precoating and postdraw coating materials.

#### 4. Bundle Drawing Studies

Bundle drawing studies will continue along the parallel routes of redrawing in a glassy matrix and redrawing in a metallic matrix.

Drawing in a glassy matrix using both soft glass coated E

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glass fibers and soft glass - metal - E glass composites fibers will be continued on the basis of their demonstrated ability to produce bundles of submicron fibers with or without a metallic coating in a glassy matrix. The effect of predraw bundling conditions, coating materials and drawing conditions on redrawn fiber diameter, roundness, and inherent matrix voids will be explored.

The redrawing of fibers bundled in a metallic matrix consisting of either the most promising Ti and/or Zr doped alloy system or deposited and fired noble metals will be further explored toward the goal of producing a metallic matrix reinforced with superstrength glass fibers.

In each of these parallel efforts, the emphasis will be placed on producing a fiber - matrix - sheath system which shows the maximum promise for producing verifiable ultrastrengths in the redrawn glass fibers.

#### 5. Bundle Separation Studies

The bundled submicron fibers produced in the bundle drawing phase will be subjected to combined chemical and mechanical processing aimed at removal of sheath material for any successful metal matrix systems and matrix and sheath (if present) for the glassy matrix systems.

Since ultrafine fibers in a glassy matrix are being drawn, in a routine fashion at this time, substantial effort will be devoted to establishing matrix removal techniques to allow strength verification studies on the redrawn fibers. The major effort here will be devoted to acid etching systems combined with mechanical agitation. In these studies, microscopic examination of progressive matrix depletion and possible fiber degradation will be used to adjust the amounts and types of matrix and interfacial materials employed in the bundle drawing operations of phase 4. If the matrix can be successfully removed, the resulting bundle of submicron fibers will be evaluated in the fiber tester constructed for use in the strength studies being conducted in phase 1. The results of these tests can be coupled with a microscopic examination of total effective fiber area to arrive at a representative determination of average strength levels for the individual fibers.

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The processes being employed in this phase are being carefully chosen to permit future translation of the bundle drawing concept into production technology.

In the event that the continuing efforts to redraw in a metallic matrix result in a fiber reinforced metallic matrix contained within a glass sheath, the glass sheath will be removed by chemical techniques similar to those being employed for the glassy matrix removal efforts. The resulting composite of ultrafine glass fibers in a soft metal matrix will be evaluated in the fiber tester using the techniques of gross strength measurement and microscopic area estimation similar to those to be employed for the fibers redrawn in a glassy matrix.

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